

Experiment 64: The preparation of an α,β -unsaturated ketone via Michael and aldol condensation reactions

Your learning outcome for this lab is to complete a mini-research project and to report your results clearly and completely. You will be adapting a procedure from Experiments 41 and 42 (which we have not done) to accomplish a synthesis and isolation of two successive compounds. **You will work in assigned teams.**

Sub-goal: To write a procedure for the synthesis that maximizes product yield.

Since this is a multi-step synthesis, you must get a percent yield of **at least 75%** of the chalcone before you can complete the experiment.

I recommend doing this experiment at least twice per partner (four trials in all) – this way, you can try a variety of reaction conditions (reaction time, temperature, solvent amount, etc.) to maximize yield.

The grading will reflect both the quality of your work (including the efficient use of the time allotted for the lab) and the ability to produce a good yield. Thus, I will check to see if the second set of trials is a sensible modification based on the yields of the first set of trials.

In addition to yield, purity is important; the melting point, IR and NMR will be good tools to use here.

• **Pre-lab:**

Read: Experiment 64 (pp. 533 – 536), Experiments 41 and 42 (pp. 339 – 347)

Prepare for class on Monday, May 7: “Purpose” and “Materials and methods”. The purpose of this experiment is straightforward: as the title suggests, you will make an α,β -unsaturated ketone via Michael and aldol condensation reactions.

From page 534, your team will *be assigned* an aldehyde to begin the synthesis.

Below the purpose, write the balanced chemical equation, using structural formulas, for this multi-step synthesis, beginning with your chosen aldehyde.

Materials: Lay out a table along the lines of the following.

Chemical	Molar mass	Mass (g) or volume (mL) used in experiment	Moles used in experiment	Is it the limiting reagent?
Your aldehyde				
Acetophenone				

Ethyl acetoacetate				

You might also include the **density** of liquid reagents under their names.

Also include a table for expected products:

Product	Moles expected	Mass of product expected (g)	Expected melting point (°C)
chalcone			
Final product			

Pre-lab exercise: Fill in as much of the previous two tables as you can, so that you can set the benchmark for a successful experiment. Your admission into lab is this set of completed tables.

Procedure: You will modify the procedure given in Experiments 41 and 42. You could start off by photocopying the lab procedures from those experiments and then adding notes in the margin as parts are modified.

Data section: Record observations about the color/heat change/gas evolution of the reaction mixture related to which step in the procedure the change occurred. In addition, you should record relevant masses and melting points.

Finally, your IR and NMR spectra should be taped to this section.

• **Post-lab:**

none

• **Lab Result Report: (Due Monday, May 21, in class)**

Note: Only one report is required from each team, and all members of the team will earn the same grade.

In writing your report, consider that your audience is Professors Pavia, Lampman, Kriz and Engel. You are submitting a procedure for possible inclusion in the fifth edition of PLKE.

(5 points) photocopy all of your team members' procedure and data sections (including spectra) only, and attach this to the back of your report.

The report has **four** sections:

Introduction (10 points) — What is the theory behind the experiment? A good start would be to modify the existing introductory section on pages 533 and 534 of PLKE to be specifically for your starting aldehyde.

Procedure (10 points) — What should the student do? Write a detailed procedure in the style of PLKE, using the procedure your team found has the highest and most pure yield. You may use some of the phrases from the textbook without fear of plagiarism. Unlike the way you may have written procedures up to this point, write this procedure for someone else to follow.

Experimental results (10 points) — What happened? Present the results of all of the syntheses for all of your team, including and especially the *unsuccessful* trials. You can write this in any form you think appropriate (PLKE does not provide a template for this), but it should be clear and complete. One approach would be to begin with an explanation of your thinking behind how the first set of trials' reaction conditions were chosen, and why you changed the particular variables for the second set of trials. All the trials' results can then be formatted into a table that shows the differences in reaction conditions that made each trial unique.

Conclusion (10 points) — Is the procedure ready for a class of 239 students to test? If this sort of testing is to be done, should the authors be looking at specific parts of the procedure that still need work or cause trouble? What do you suggest the authors do next to this experiment if more work is needed?